

PREPARATION AND THERMOELECTRIC PROPERTIES OF $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ ALLOYS

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Abstract

The preparation and characterization of the binary arsenopyrite compounds CoSb_2 and IrSb_2 and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ alloys is reported. Single crystals of CoSb_2 were grown by the vertical gradient freeze technique from solutions rich in antimony. Polycrystalline samples of IrSb_2 and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ alloys were prepared by hot-pressing of prereacted elemental powders. Samples were investigated by X-ray diffractometry, microprobe analysis and density measurements. It was found that a range of solid solution exist in the system $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ for $0.1 \leq x \leq 0.8$. Samples were also characterized by high temperature electrical resistivity, Seebeck coefficient and thermal conductivity measurements. All materials have p-type conductivity and are semiconductors. A band gap of about 0.98 eV was calculated for IrSb_2 . Preliminary measurements of the thermoelectric properties of these materials showed that their potential for thermoelectric applications is limited.

Keywords: A. intermetallic compounds, A. semiconductors, B. crystal growth, D. transport properties.

1. introduction

A large number of compounds with the pyrite, marcasite and arsenopyrite structure has been reported in the literature. These three structure type are closely related to each other [1]. Most of these compounds are semiconductors [1]. The structure of most these compounds has been well characterized but data about their electrical and thermoelectric properties is limited, especially for the arsenopyrite compounds. The arsenopyrite structure was first established for a mineral of FeAsS by Buerger [2]. However, CoSb_2 is generally accepted as the prototype for this class of compounds [3]. Binary arsenopyrite compounds are formed by combination of Co, Rh and Ir with P, As, Sb and Bi with the exception of CoP_2 and CoBi_2 . Many ternary compounds with the arsenopyrite monoclinic structure have also been reported in the literature [1].

Our interest in these compounds resulted from a search for new thermoelectric materials. Very little is known about the thermoelectric properties of arsenopyrite compounds and we report in this paper on the preparation and thermoelectric properties of the binary compounds IrSb_2 , CoSb_2 and their solid solutions. CoSb_2 and IrSb_2 are isostructural compounds and recent structural data for these compounds can be found in reference [3]. In a recent study of the high temperature behavior of the compounds CoAs_2 and CoSb_2 by magnetic, electrical and calorimetric measurements on small single crystals prepared by a chemical vapor technique, Siegrist and Mulliger found that these compounds undergo a transformation from the arsenopyrite to marcasite structure at 527°C and 371°C for CoAs_2 and CoSb_2 , respectively [4]. They also estimated a band gap of about 0.17 eV for CoSb_2 from high temperature resistivity measurements. This is in good agreement with a value of 0.2 eV determined by Dudkin and Abrikosov using the same type of measurements [5]. Siegrist and Mulliger concluded that the low-temperature arsenopyrite phase of CoSb_2 is semiconducting whereas the high-temperature marcasite phase has a metallic behavior. They measured a p-type Seebeck coefficient of about $40 \mu\text{V}\cdot\text{K}^{-1}$ on their crystals. Although some data are available for CoSb_2 , a full characterization of its thermoelectric properties was not accomplished up to now and no data are available about the electrical properties of IrSb_2 to the best of our knowledge. We prepared samples of IrSb_2 , CoSb_2 and their alloys and measured some of their thermoelectric properties. The results are presented in this paper.

2. Experimental

Crystals of CoSb_2 were grown by the gradient-freeze technique. The compound CoSb_2 forms peritectically at 929°C [6]. The Co-Sb phase diagram shows that the growth of CoSb_2 can be initiated from Sb rich melts between 83 and ~ 90 at.% Sb [6]. A two-zone furnace was used and a thermal baffle was introduced between the upper and the lower zone in order to prevent air convection. An opening of about 12.5 mm in diameter was made in the center of this baffle to introduce the melt container. Details about the furnace used for the growth can be found in reference [7]. The growth was conducted in a sealed quartz ampoule which was stationary during the growth. The temperature close to the upper part of the thermal baffle was controlled by a temperature programmer controller and a second temperature controller maintained a constant temperature gradient between the upper and the lower zones of the furnace. The furnace was calibrated and temperature gradients as high as $125^\circ\text{C}/\text{cm}$ were obtained near the interface. The growth process was carried out by lowering the temperature of the furnace and the temperature gradient could be adjusted by changing the difference in the temperatures maintained between the two zones. Co (99.99%/0) and Sb (99.9999%/0) were introduced into pointed quartz ampoules, coated with graphite, and sealed under vacuum. Two different nominal compositions of the melt were used: 85 and 87 at.% Sb and the total charge was about 30 g. A temperature gradient of about $50^\circ\text{C}/\text{cm}$ was maintained at the growth interface and the growth rate was about $0.7^\circ\text{C}/\text{hour}$.

It was found in a recent investigation of the Ir-Sb phase diagram that the compound IrSb_2 decomposes peritectically at about 1475°C [8]. Because of the high temperatures required for the growth of IrSb_2 crystals from the melt, the preparation of polycrystalline samples of IrSb_2 and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ solid solutions was preferred. Single

phase, polycrystalline samples were prepared by direct reaction of the elements. Iridium (99.9s%), cobalt (99.99%) and antimony (99.9999%) powders were mixed in stoichiometric ratio in a plastic vial before being loaded in a steel die where they were compressed into a dense cylindrical pellet. The pellet was sealed under vacuum in a quartz ampoule which was heated for several days at a temperature between 600 to 1000°C. The product was then removed from the ampoule, crushed, ground in an agate mortar, loaded again in a quartz ampoule and heated for several days at a temperature between 880 and 1000°C. The exact duration and temperature of the annealing is reported in the result section. Products of the annealing were removed from the ampoules and analyzed by X-ray diffractometry (XRD). Dense samples about 10 mm long and 6.35 mm in diameter were prepared by hot -pressing of the prereacted powders in graphite dies. The hot-pressing was conducted at a pressure of about 20,000 psi and at a temperature between 800 and 900°C for about 2 hours.

XRD analysis was performed at room temperature on a Siemens D-500 diffractometer using the Cu-K α radiation. Small additions of Si powders were made to the samples as an internal standard, Powder X-ray patterns were taken with scan steps of $2\Theta=0.05^\circ$ and counting time of 3 s. Hot-pressed samples were polished using standard metallographic techniques and were investigated using an optical microscope with and without light polarization or Nomarski contrast to observe their quality and homogeneity. Microprobe analysis (MPA) was performed on selected samples to determine their atomic composition using a JEOL JXA-733 electron superprobe operating at 20 kV of accelerating potential and 25×10^{-9} A of probe current. Pure elements were used as standards and X-ray intensity measurements of peak and background were conducted by wavelength dispersive spectrometry. Mass densities were determined using the immersion technique and toluene as the liquid.

The electrical and thermal transport properties of the samples investigated were measured between room temperature and about 600°C. The electrical resistivity and the Hall coefficient were measured by the van der Pauw method using tungsten probes located on the top surface of the sample as close as possible to the sample's edge (typically a 1 mm thick, 6.35 mm diameter slice) [9]. The Seebeck coefficient measurement was conducted by creating a variable temperature difference across the sample and measuring the corresponding linear variation of its thermoelectric voltage [10]. Large samples are usually measured by this technique but samples as thin as 1 mm can also be accommodated. The thermal diffusivity and the heat capacity of selected samples was measured by a flash diffusivity technique [11].

3. Results for CoSb₂ and IrSb₂

Several ingots of CoSb₂ were successfully grown and, as expected, the grown ingots were composed of two parts: the lower part corresponding to the arsenopyrite compound and the upper part corresponding to a Sb-rich eutectic. The lower part of the ingots was cut from the rest of the ingot using a diamond saw and subjected to further analysis. The density of the entire lower portions of the ingots were found to be about 99.6 % of the theoretical density of CoSb₂, 8.34 g.cm⁻³. MPA showed that the samples were single phase with a composition close to 1:2, in agreement with the results of

Kjekshus who found that CoSb_2 does not have appreciable stoichiometric deviations [3]. Ingots of CoSb_2 were typically composed of a few large grains (~5 mm) but single crystals about 10 mm in diameter and 1 mm thick were also obtained. Samples in the form of disks about 1 mm thick and 10 mm in diameter were cut from the single crystal part of the ingots for transport property measurements. The results of these measurements are presented in the following paragraph. The density of the IrSb_2 hot-pressed samples was found to be about 95% of the theoretical density, 11.06 g.cm^{-3} . MPAs of the hot-pressed samples showed that the samples were single phase with a composition close to 1:2. Samples about 6.35 mm in diameter and 1 mm thick were cut from the hot-pressed bars for thermoelectric measurements.

The room temperature properties of one hot-pressed IrSb_2 sample and several CoSb_2 samples cut from two ingots grown from two different nominal compositions are reported in Table 1. All samples have p-type conductivity at room temperature. A maximum Hall mobility value of about $150 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$ was measured on a CoSb_2 crystal with a carrier concentration of $1.15 \cdot 10^{20} \text{ cm}^{-3}$ which is relatively high at this doping level. Very little variation is observed in carrier concentration for CoSb_2 samples obtained from ingots grown from different nominal melt compositions. This result supports the idea that CoSb_2 is a compound with small stoichiometric deviations and the carrier concentration of the samples does not vary with the temperature of crystallization. Because of the high doping level but also the relatively high Hall mobility values, the electrical resistivity values of the CoSb_2 samples are low. The IrSb_2 hot-pressed sample has also a p-type conductivity but a much lower doping level and Hall mobility, resulting in quite a large electrical resistivity value.

The results of high temperature electrical resistivity, Hall mobility, Seebeck coefficient and thermal conductivity measurements on two CoSb_2 crystals (1NG2 and 2NB 10) and one hot-pressed IrSb_2 sample (1NF8HP) are shown in figures 1, 2, 3, 4 and 5. The variations of the electrical resistivity (ρ), Hall mobility (μ), Seebeck coefficient (α) and thermal conductivity (λ) with temperature consistently show a transition in the properties of CoSb_2 samples at a temperature of about 370°C . This transition corresponds to the transition from the arsenopyrite structure at low temperature to the marcasite structure above this temperature which has been described in details by Kjekshus and Rakke [12]. Our transition temperature for CoSb_2 is in excellent agreement with a transition temperature of 371°C reported by Siegrist and Mulliger [4] and 377°C by Kjekshus and Rakke [12]. For IrSb_2 , no transition was observed up to 600°C , in agreement with the results of Kjekshus and Rakke who found that IrSb_2 maintains the monoclinic structure up to a maximum temperature of about 1027°C [12]. Thermal and electrical properties of CoSb_2 show that the low-temperature arsenopyrite phase behaves as a heavily doped semiconductor. The resistivity increases up to the transition temperature and the doping level of the crystals was too high to observe any intrinsic behavior and estimate a band gap. The Hall mobility and Seebeck coefficient decrease up to the transition temperature. The high-temperature marcasite structure has a distinct metallic behavior: very low Hall mobility, low Seebeck coefficient and increasing thermal conductivity with temperature. The thermal conductivity of CoSb_2 is about $118 \text{ mW.cm}^{-1} \cdot \text{K}^{-1}$ at room temperature and decreases to a minimum value of about $50 \text{ mW.cm}^{-1} \cdot \text{K}^{-1}$ at the transition temperature.

Figure 1 shows that an intrinsic behavior is observed at high temperatures in the variations of the electrical resistivity of the p-type IrSb_2 sample. A band gap of about 0.98 eV was calculated for IrSb_2 from the quasi-linear variations of the electrical resistivity at high temperatures. The Hall mobility and the Seebeck coefficient of the p-type sample (1 NF8HP) increase with temperature, indicating that mixed conduction occurs at low temperatures. The thermal conductivity decreases with temperature from a room temperature value of about $105 \text{ mW.cm}^{-1}.\text{K}^{-1}$ to a minimum value of about $50 \text{ mW.cm}^{-1}.\text{K}^{-1}$ between 600 and 700°C . These relatively high thermal conductivity values limit the thermoelectric figure of merit (Z) of IrSb_2 defined as $Z = \alpha^2/\rho\lambda$. Although no efforts were made to optimize the carrier concentration in order to decrease the electrical resistivity, it seems unlikely that high figures of merit can be achieved for IrSb_2 . The same conclusions can be drawn for CoSb_2 which, despite the good Hall mobility obtained for this compound, has also a rather large thermal conductivity. Reduction in thermal conductivity might be achieved in solid solutions between isostructural compounds. We investigated the existence and properties of $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ solid solutions. The results are presented in the following section.

4. Results for $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ solid solutions

Alloys in the composition range 0-100 mole% CoSb_2 were prepared. The preparation conditions and the results of the characterization of the samples are summarized in Table 2. MPA and X-ray analysis showed that a partial solid solution exist in the system $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ for $0.1 \leq x \leq 0.8$. X-ray analysis of samples with 50 and 80 mole% of CoSb_2 showed that they were multiphase and contained two arsenopyrite phases: one IrSb_2 -rich and the second one CoSb_2 -rich. Figure 6 shows the X-ray pattern for IrSb_2 and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ alloys with $x = 0.9, 0.8$ and 0.1 . A shift in the peak position around $2\theta = 49^\circ$ can be observed for IrSb_2 -rich alloys and is an indication of the solid solution formation. Only very slight shifts can be observed for some angles which indicates that the structure is distorted only in some crystallographic directions. The pattern for the alloy with $x = 0.1$ is consistent with the pattern of CoSb_2 and shows a shift in the peak position,

We investigated the thermoelectric properties of alloys within this range of composition. Three samples with 10, 20 and 90 mole% CoSb_2 were prepared and hot-pressed. The experimental densities measured on the hot-pressed samples are listed in Table 2 and are close to the calculated theoretical density. The results of MPA of these samples showed that the sample were single phase and confirmed the existence of a range of solid solutions for $0.1 < x \leq 0.8$ found by XRD analysis. The room temperature properties of the alloys are summarized in Table 2. All the alloys have p-type conductivity. The trends observed for the binary compounds can also be seen for the alloys: the IrSb_2 -rich have relatively large electrical resistivity and low Hall mobility values whereas the CoSb_2 -rich alloy has a low electrical resistivity value and a good Hall mobility value. The results of high temperature electrical resistivity, Hall mobility, Seebeck coefficient and thermal conductivity measurements on the CoSb_2 -rich alloy (2A15) and two IrSb_2 -rich alloys (3A16 and 3A17) are shown in figures 1, 2, 3 and 4,

respectively. The CoSb_2 -rich alloy behaves similarly to CoSb_2 . The phase transition (arsenopyrite to marcasite) can be observed at a temperature of about 350°C in the variations of the Seebeck coefficient and Hall mobility with temperature. The Seebeck coefficient values are relatively low. The IrSb_2 -rich alloys behave quite differently than the compound IrSb_2 . Effects of mixed conduction are not as strong at low temperatures in the alloys because of their higher doping level. The alloys have much larger Seebeck coefficient values than IrSb_2 but the intrinsic domain seems to be shifted towards lower temperatures when the material contains more CoSb_2 . This might be related to a decrease in the band gap of the alloys from IrSb_2 (0.98 eV) to CoSb_2 (-0.23 eV) [4]. The power factor values (α^2/ρ) for the CoSb_2 -rich solid solution are low due to the low Seebeck coefficient of the samples. The power factor values of the two IrSb_2 -rich alloys increase with temperature and a maximum value of about $11 \mu\text{W}\cdot\text{cm}^{-1}\text{K}^{-2}$ was obtained for the alloy with 10 mole% of CoSb_2 at 600°C .

Significant improvements in the figure of merit by forming solid solutions are possible because of the drop in lattice thermal conductivity when the drop in carrier mobility is not too important. The two IrSb_2 -rich alloys were measured for thermal diffusivity and heat capacity from room temperature to about 600°C . Combining these two measurements, the calculated thermal conductivity of these samples are shown in Figure 5. Results show a very large drop in thermal conductivity compared to the values for the compounds IrSb_2 and CoSb_2 . The thermal conductivity for both alloys is almost temperature independent which indicates a very strong point defect scattering. The thermal conductivity for the alloy with 20 mole% of CoSb_2 is about $28 \text{ mW}\cdot\text{cm}^{-1}\cdot\text{K}^{-1}$, significantly lower than for the alloy with 10 mole%. These values are comparable to state-of-the-art high temperature thermoelectric materials such as SiGe alloys [13]. A maximum ZT value of about 0.2 was obtained for the alloy with 10 mole% of CoSb_2 at a temperature of about 600°C . Preliminary measurements on alloys indicate that their thermoelectric potential is limited by their low Hall mobility for IrSb_2 -rich alloys and low Seebeck coefficient values for the CoSb_2 -rich alloys. The thermoelectric figure of merit of these materials might slightly be improved by optimization of their doping level of these alloys but ZT values higher than state-of-the-art thermoelectric materials are unlikely to be achieved.

Conclusion

Single crystals of CoSb_2 were grown and polycrystalline samples of IrSb_2 were prepared by hot-pressing of prereacted elemental powders of Ir and Sb. X-ray and microprobe analysis of alloys in the system $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ showed that there is a range of solid solution for $0.1 \leq x \leq 0.8$. The thermoelectric potential of CoSb_2 , IrSb_2 and their alloys was investigated by high temperature electrical resistivity, Seebeck coefficient and thermal conductivity measurements. It was found that all the samples investigated were semiconductors and a band gap of about 0.98 eV was calculated for IrSb_2 . CoSb_2 crystals are characterized by a relatively high mobility, low Seebeck coefficient and a relatively large thermal conductivity. Thermoelectric property measurements of CoSb_2 crystals also revealed a transition from the arsenopyrite to the marcasite structure at a temperature of about 370°C , in good agreement with previous literature results. No transformation was observed for the IrSb_2 hot-pressed samples up to 600°C . The IrSb_2 samples are

characterized by a low carrier mobility and high thermal conductivity. $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ alloys behave similarly to the closest binary compound but have much lower thermal conductivity due to increased point defect scattering. Preliminary measurements of the thermoelectric properties of these arsenopyrite materials showed that their thermoelectric potential is limited.

Acknowledgments

The work described in this paper was carried out by the Jet Propulsion Laboratory/California Institute of Technology, under contract with the National Aeronautics and Space Administration. The author would like to thank Dr. Jean-Pierre Fleurial and Dr. Alex Borshchevsky for useful discussions, Danny and Andy Zoltan for thermoelectric properties measurements, Paul Carpenter for microprobe analyses and Jim Kulleck for XRD analyses.

References

1. Hulliger F. and Mooser E., *J. Phys. Chem. Solids* **26**, 429(1965).
2. Buerger M. J., *Z. Krist.* **95**,83 (1936).
3. Kjekshus A., *Acta Chem. Stand* **25**,2(1971).
4. Siegrist T. and Hulliger F., *J. Solid State Chem.* **63**,23 (1986).
5. Dudkin L. D. and Abrikosov N. Kh., *Russ. J. Inorg. Chem.* **2**,325 (1957).
6. Feschotte P. and Lorin D., *J. Less-Common Metals* **155**,255 (1989).
7. Borshchevsky A., Caillat T. and Fleurial J. -P., *NASA Tech. Briefs* **18**,3,68 (1994).
8. Caillat T., Borshchevsky A, and Fleurial J. -P., *J. of Alloys and Compounds* **199**,207 (1993).
9. McCormack J. A. and Fleurial J. -P., in *Modern Perspectives on Thermoelectrics and Related Materials*, MRS Symp. Proc. 234, 135, (Materials Research Society, Pittsburgh, Pennsylvania 1991).
10. Wood C., Zoltan D. and Stapfer G., *Rev. Sci. Instrum.* **S6**, 5, 719(1985).
11. Vandersande J. W., Wood C., Zoltan A. and Whittenberger D., *Thermal Conductivity*, Plenum Press, New York, 445 (1988).
12. Kjekshus A. and Rakke T., *Acta. Chem. Stand Ser. A* **31**, 517(1977).
13. Vining C. B., *J. Appl. Phys.* **69**, 1,331 (1991).

Tables captions

- Table 1. Some properties of the compounds IrSb_2 and CoSb_2 samples at room temperature.
- Table 2. Some properties of $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ alloys at room temperature. The theoretical density of the $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ alloys was calculated assuming a linear variation of the density between the binary compounds.

Figure captions

- Figure 1. Electrical resistivity versus inverse temperature IrSb_2 sample (1NF8HP) and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ samples with $x=0.9$ (3A16) and $x=0.8$ (2A17).
- Figure 2. Electrical resistivity versus inverse temperature for CoSb_2 samples (1NG2 and 2NB10) and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ sample with $x=0.1$ (2A15).
- Figure 3. Hall mobility versus temperature for CoSb_2 samples (1NG2 and 2NB10), IrSb_2 sample (1NF8HP) and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ samples with $x=0.9$ (3A16), $x=0.8$ (2A17) and $x=0.1$ (2A15).
- Figure 4. Seebeck coefficient versus inverse temperature for CoSb_2 samples (1NG2 and 2NB10), IrSb_2 sample (1NF8HP) and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ samples with $x=0.9$ (3A16), $x=0.8$ (2A17) and $x=0.1$ (2A15).
- Figure 5. Thermal conductivity versus inverse temperature for CoSb_2 (1NG2), IrSb_2 (1NF8HP) and $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ samples with $x=0.9$ (3A16) and $x=0.8$ (2A17).
- Figure 6. X-ray diffraction pattern for $\text{Ir}_x\text{Co}_{1-x}\text{Sb}_2$ alloys with $x=1, 0.9, 0.8, 0.1$ and 0. For clarity, selected angles are shown only.

Sample	Nominal Composition (at%)		Preparation method	Type	Room temperature properties				
					n (cm^{-3})	μ ($\text{cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$)	ρ ($\text{m}\Omega \cdot \text{cm}$)	α ($\mu\text{V} \cdot \text{K}^{-1}$)	λ ($\text{mW} \cdot \text{cm}^{-1} \cdot \text{K}^{-1}$)
CoSb ₂	co	Sb							
I-NG2	18	82	Bridgman	p	1.24E+20	145.7	0.34	26	118
3-NG2	18	82	Bridgman	p	1.60E+20	120.0	0.32	-	
3-NG2	18	82	Bridgman	p	1.58E+20	131.0	0.30	-	
1-NB10	13	87	Bridgman	p	1.24E+20	136.4	0.37	33	-
2-NB10	13	87	Bridgman	p	1.33E+20	1301	0.37	30	
3-NB10	13	87	Bridgman		1.16E+20	150.4	0.36	38	-
IrSb ₂	Ir	Sb							
1NF8HP	33.33	66.67	Powder metallurgy 1000°C/1 day + 900°C/ 7 days	p	3.68E+18	26.0	65.35	60	105

Table 1

Sample	CoSb ₂	Preparation Method	Heat treatment conditions	X-ray results	Experimental density (g cm ⁻³)	Theoretical density (g cm ⁻³)	Type	Room temperature properties			
					(g cm ⁻³)	(g cm ⁻³)		n	μ	ρ	α
A15	90	cold-press + anneal	600°C/5 days + 880°C/2 days	CoSb ₂ -rich arsenopyrite phase	8.50	8.51	p	7.29E+19	104.80	0.82	74
A18	80	cold-press + anneal	900°C/4days + 900°C/6 days	IrSb ₂ +CoSb ₂ -rich arsenopyrite phases	-	-	-	-	-	-	-
A19	50	cold-press + anneal	900°C/4days + 900°C/6 days	IrSb ₂ +CoSb ₂ -rich arsenopyrite phases	-	-	-	-	-	-	-
A17	20	cold-press + anneal	900°C/4days + 1000°C/4 days	IrSb ₂ -rich arsenopyrite phase	10.23	10.52	p	1.53E+19	0.75	539.60	125
A16	10	cold-press + anneal	900°C/4days + 1000°C/2 days	IrSb ₂ -rich arsenopyrite phase	10.46	10.79	p	2.55E+18	17.4	143.47	320
IrSb ₂	0				11.06	11.06					

Table 2

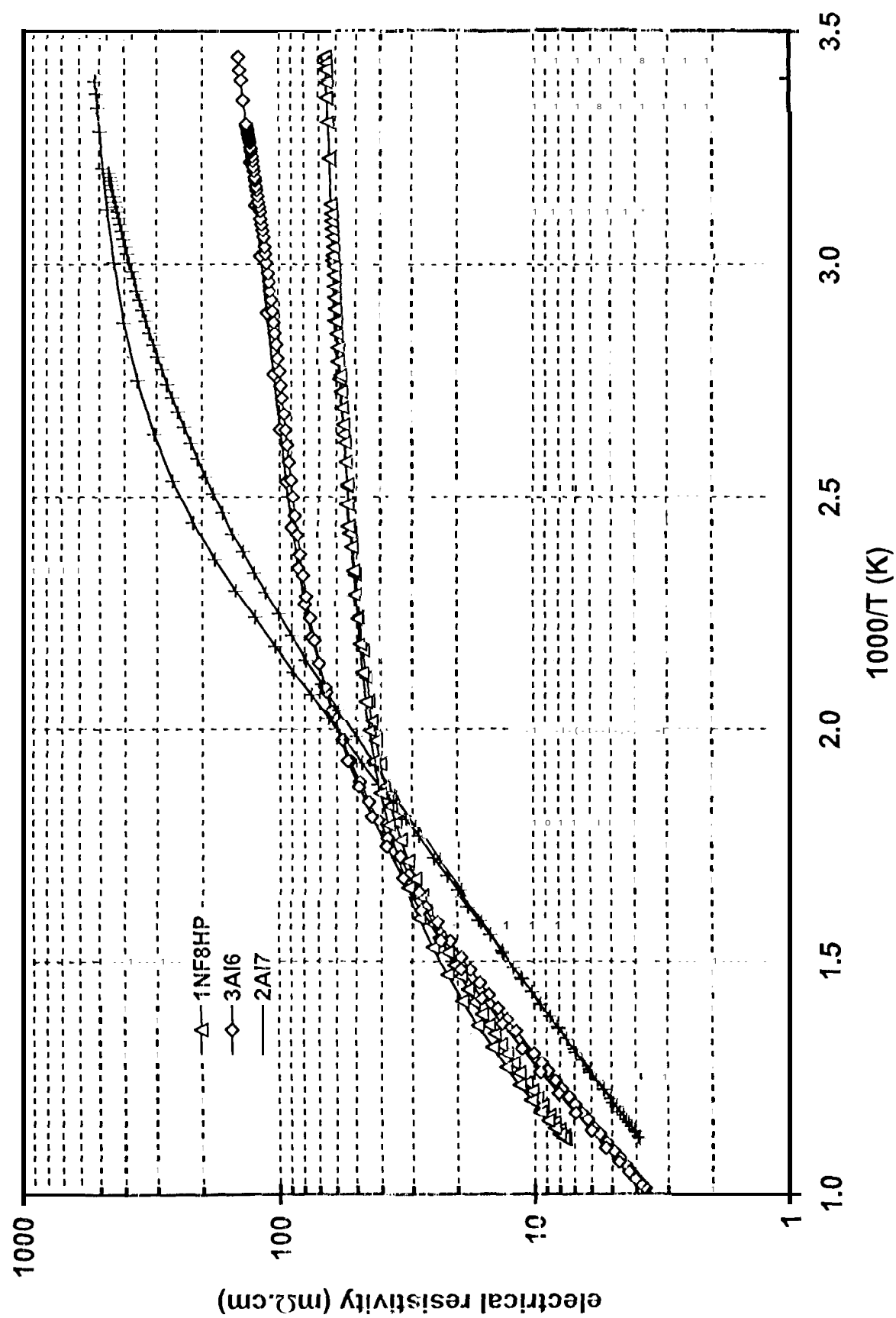


Figure 1

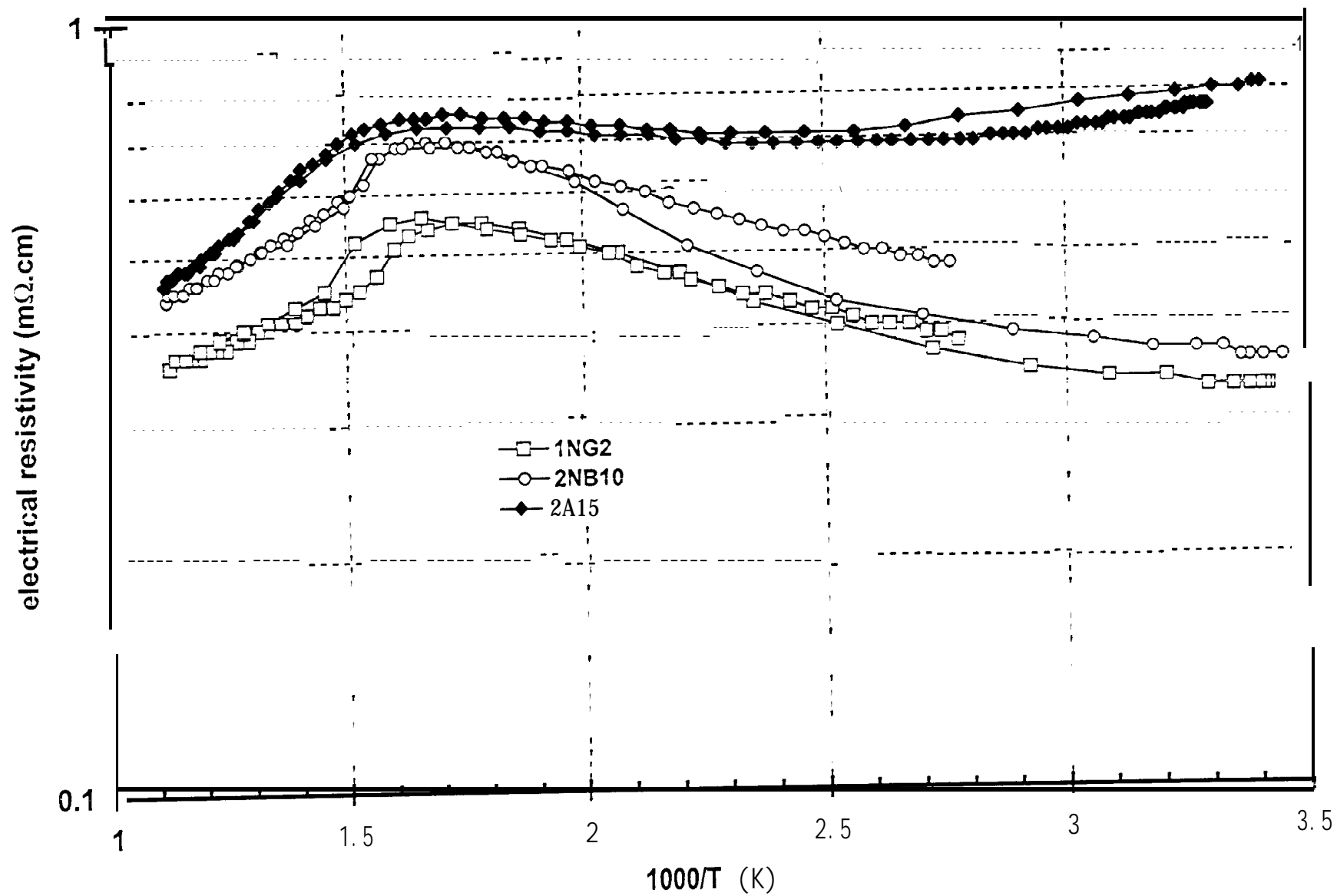


Figure 2

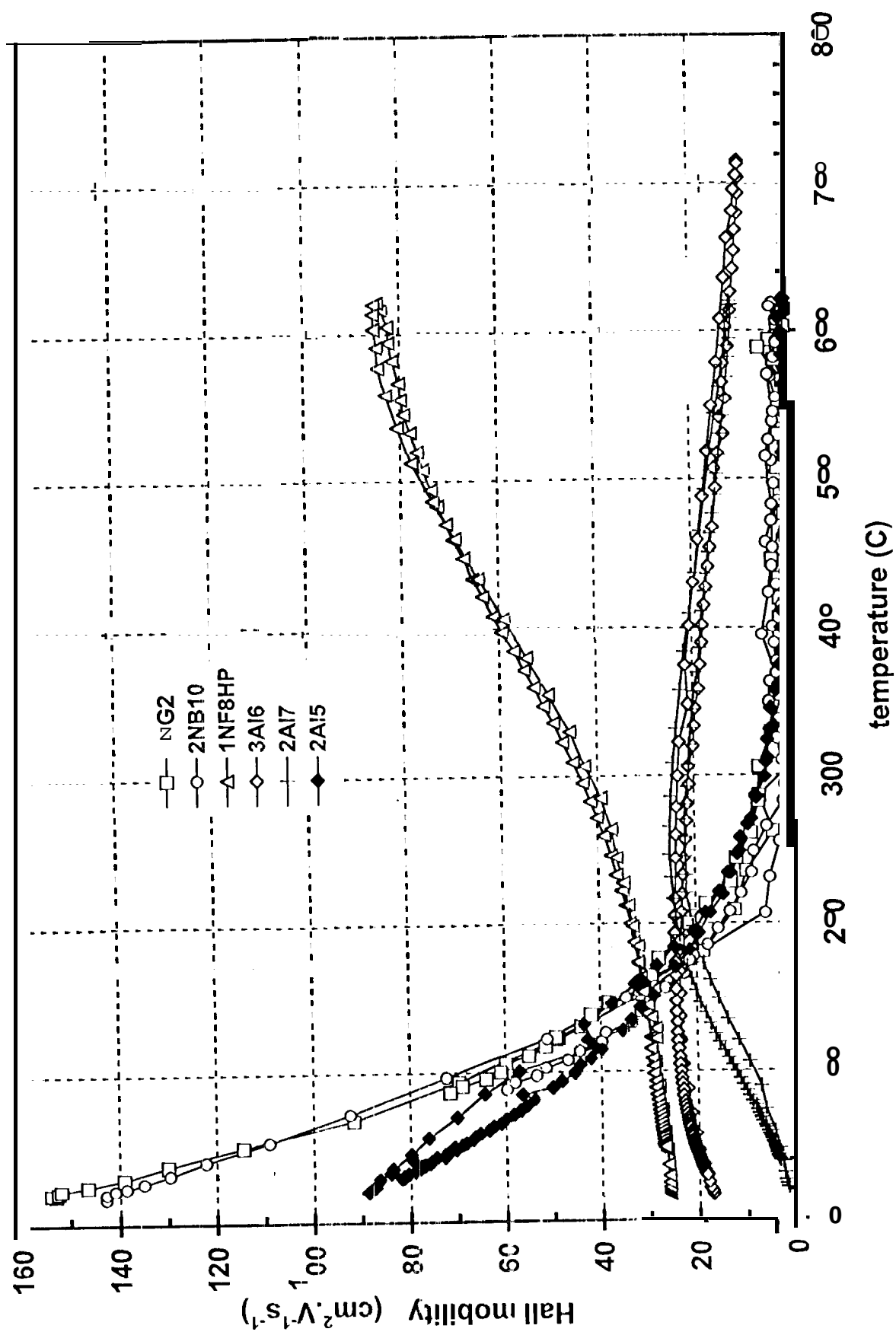


Figure 3

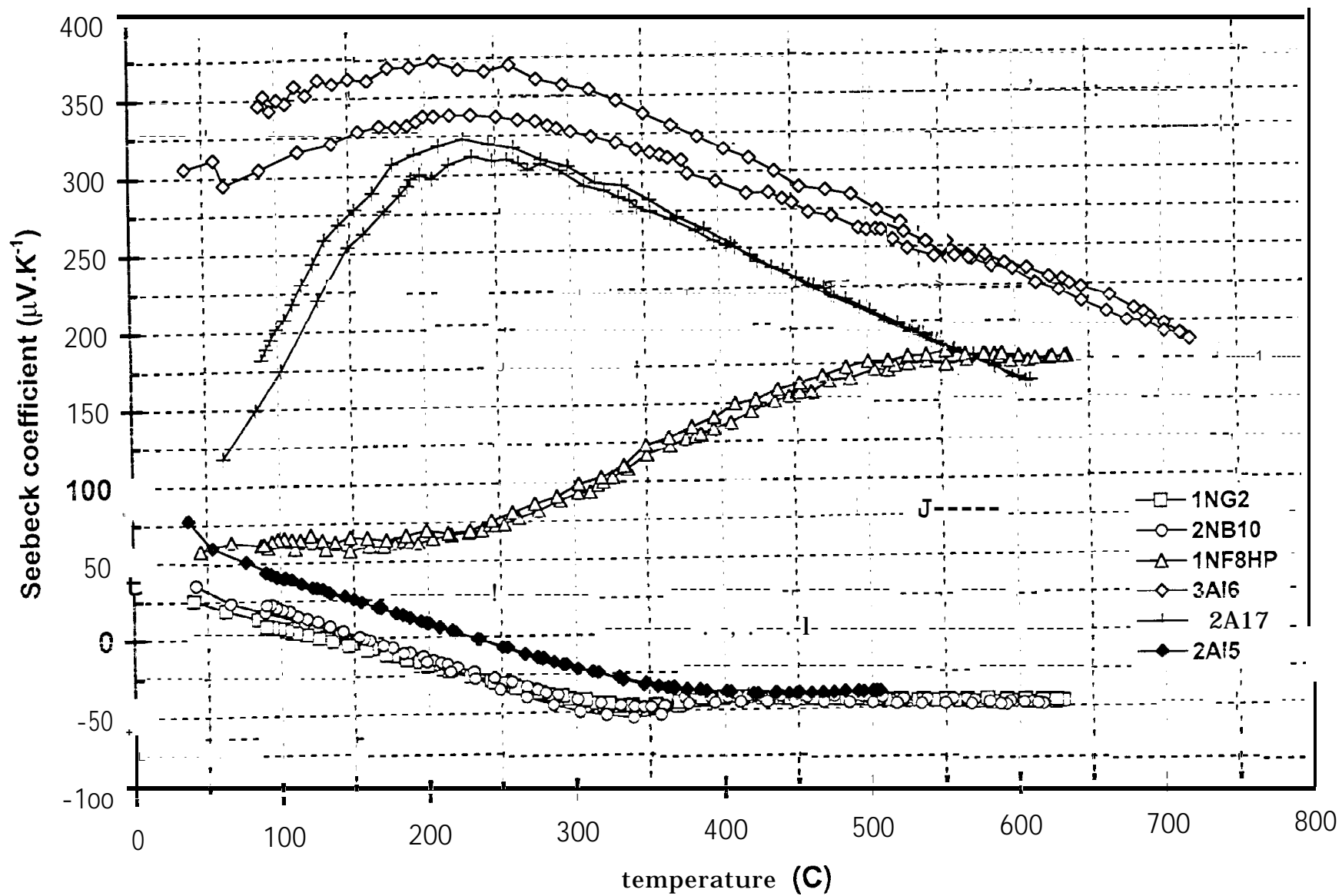


Figure 4

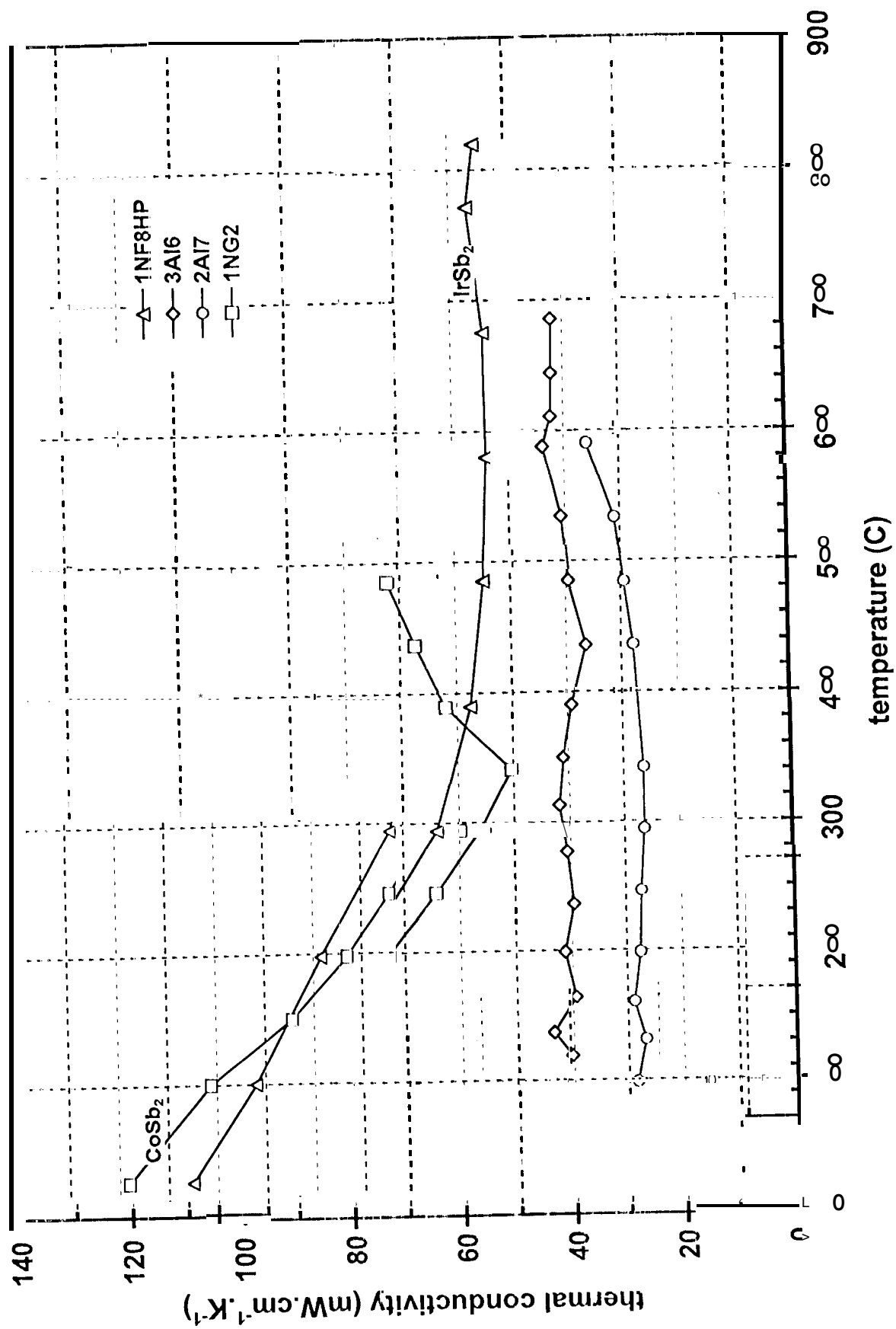


Figure 5

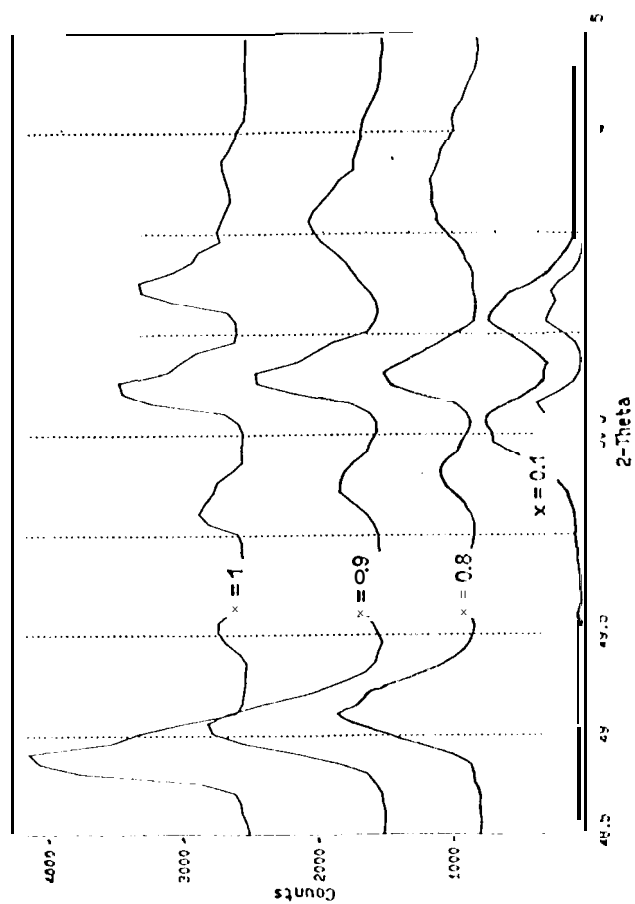


Figure 6